

# Nitrogen, nitrite, colorimetry, diazotization, automated-segmented flow

Parameter and Code:  
Nitrogen, nitrite, dissolved, I-2540-90 (mg/L N): 00613

## 1. Application

This method is used to analyze samples of surface, domestic, and industrial water and brines containing from 0.01 to 1.0 mg/L of nitrite-nitrogen. Concentrations greater than 1.0 mg/L must be diluted. This modified method was implemented in the National Water Quality Laboratory in March 1988.

## 2. Summary of method

Nitrite ion reacts with sulfanilamide under acidic conditions to form a diazo compound which then couples with N-1-naphthylethylenediamine dihydrochloride to form a red compound, the absorbance of which is measured colorimetrically (Shinn, 1941; Bendschneider and Robinson, 1952; Fox, 1979, 1985; Pai and others, 1990).

## 3. Interferences

Concentrations of potentially interfering substances generally are negligible. For specific details of inorganic and organic compounds that interfere with the reaction, see Norwitz and Keliher (1985, 1986) as well as more general information from the American Society for Testing and Materials (1991).

## 4. Apparatus

4.1 *Alpkem rapid flow analyzer (RFA)*, consisting of sampler, peristaltic pump, analytical cartridge, colorimeter, data station, and printer.

4.2 With this equipment, the following operating conditions are satisfactory for the range from 0.01 to 1.0 mg/L N:

Flow cell	10 mm
Wavelength	540 nm (or 520 nm)
Sampling rate	90 per hour
Sample time	24 seconds
Wash time	16 seconds
Pecking	ON
Damp (RC)	I second

## 5. Reagents

5.1 *Color reagent*: Add 200 mL concentrated phosphoric acid (sp gr 1.69) And 20 g sulfanilamide to about 1,500 mL demineralized water. Dissolve completely (warm if necessary). Add 1.0 g N-1-naphthylethylenediamine dihydrochloride and dissolve completely. Dilute to 2 L with demineralized water. Add 1 mL Brij-35 solution. Store in an amber bottle and refrigerate. The reagent is stable for about 1 month.

5.2 *Nitrite-nitrogen standard solution I*, 1.00 mL = 0.100 mg NO<sub>2</sub>-N:: Dissolve 0.6706 g KNO<sub>2</sub>, dried overnight over sulfuric acid, in demineralized water and dilute to 1,000 mL. This solution and the following nitrite standard solutions (paragraphs 5.3 and 5.4) are not stable indefinitely; their concentrations need to be checked frequently.

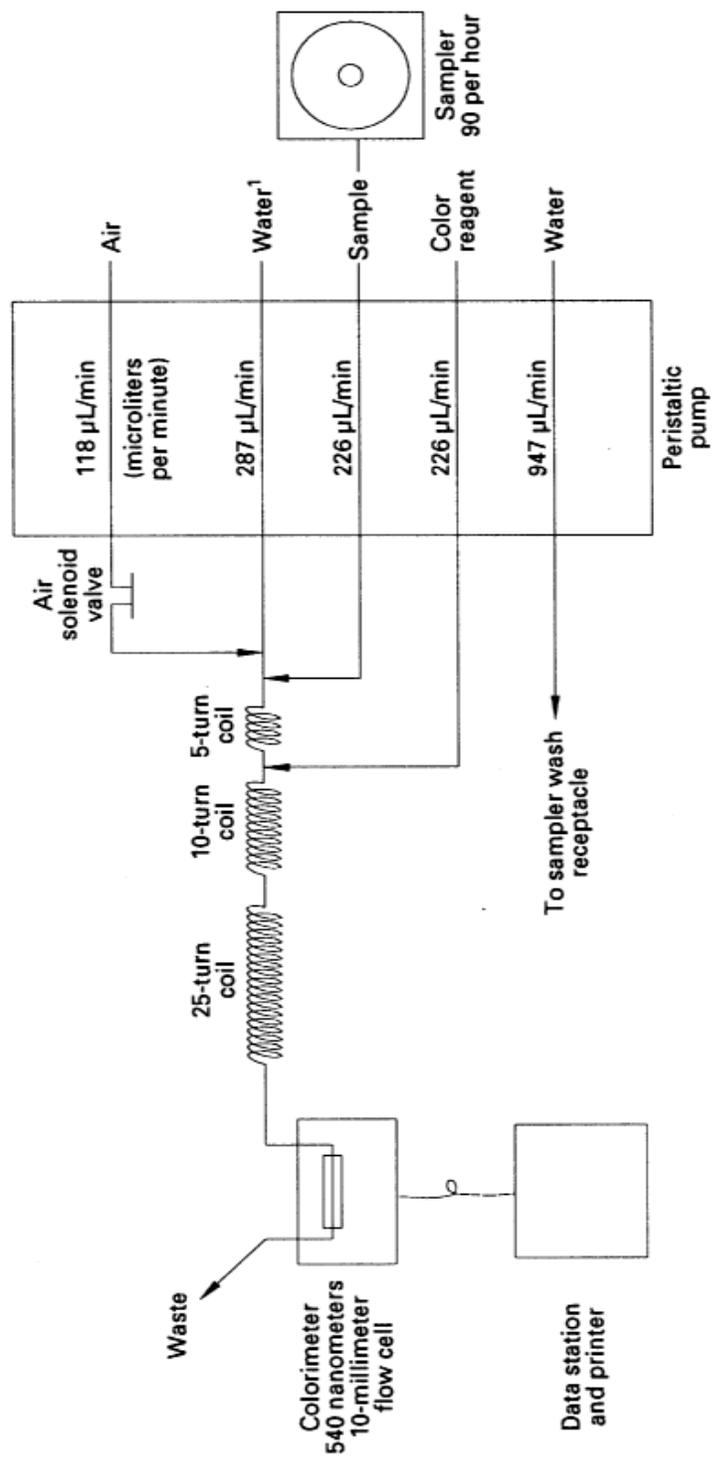
5.3 *Nitrite-nitrogen standard solution II*, 1.00 mL = 0.001 mg NO<sub>2</sub>-N: Dilute 10.0 mL nitrite-nitrogen standard solution I to 1,000 mL with demineralized water.

5.4 *Nitrite-nitrogen working solutions*: Prepare a blank and 250 mL of a series of working solutions by appropriate dilution of nitrite-nitrogen standard solution II and appropriate working solutions, as listed in the following table. If the samples to be analyzed are preserved, the nitrite-nitrogen working solutions need to contain an equivalent concentration of the same preservative.

Working solution No.	Solution added (mL)	Solution used	Nitrite-nitrogen concentration (mg/L)
1	250	Standard solution II	1.00
2	125	Standard solution II	.50
3	50	Standard solution II	.20
4	25	Standard solution II	.10
5	25	Working solution No. 2	.05
6	25	Working solution No. 4	.01

## 6. Procedure

6.1 Set up manifold (fig. 6).



<sup>1</sup> Contains 0.5-milliliter Brij solution per liter.

Figure 6. – Nitrogen, nitrite, diazotization manifold.

6.2 Allow colorimeter recorder to warm for at least 10 minutes.

6.3 After all reagents are on line, adjust the sample output of the photometer to 5 V. Then switch the photometer to “absorbance” mode and use the reference detector “fine gain” control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986).

6.4 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.5 When the system is clear of all working solutions, determine a dwell time using the most concentrated working solution.

6.6 Place a complete set of working solutions and a blank in the first positions of the sample tray beginning with the most concentrated working solution. Place individual working solutions of differing concentrations in approximately every tenth position on the tray following the accepted protocol. Fill the remainder of each tray with samples.

6.7 Begin analysis.

## **7. Calculations**

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective nitrite-nitrogen concentration, or by using the RFA Softpac data reduction package. See operation manuals for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved nitrite-nitrogen in each sample either by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or working solution peak; the RFA software automatically corrects for baseline drift.

## **8. Report**

Report concentrations of nitrite-nitrogen, dissolved (00613), as follows: less than 1.0 mg/L, two decimals; 1.0 mg/L and greater, two significant figures.

## 9. Precision

Single operator precision for nitrite-nitrogen, as determined for natural-water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

Number of determinations	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
240	0.05	0.001	2.0
209	.03	.001	3.3
240	.26	.001	.38

## References

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- American Society for Testing and Materials, 1991, Annual book of ASTM standards, Section 11, Water: Philadelphia, v. 11.01, p. 508-515.
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